

11-01-2005

**DUPLICATE**

007 11.01.2005 107 10/531194 EP0312074

JC12 Rec PCT/PTC 13 APR 2005

1

CLAIMS

1. Method for modifying electronic properties of a sample surface to analytical ends, comprising in situ deposition of neutral cesium ( $Cs^0$ ), under ultra-high vacuum, said neutral cesium being enabled in the form of a collimated adjustable stream, said  $Cs^0$  deposition being simultaneously accompanied by a primary bombardment of said surface, in the form of at least a beam comprising electrons and/or ions or neutral atoms or groups of atoms, or by an X-ray irradiation, intended to induce a secondary emission or sputtering of particles for analysis, out of the surface, said sputtering comprising secondary electrons and/or  $Cs_x^{n+}$  and/or  $MCs_x^{n+}$  positive clusters ( $x = 1, 2$ ) and/or  $M^{m-}$  negative ions and/or  $M^{m+}$  positive ions,  $M$  being a constituent of the sample material made of an atom or a group of atoms ( $n, m$  integers), the  $Cs^0$  deposition being decoupled from the primary bombardment conditions, to provide a simultaneous optimization of deposited  $Cs^0$  concentration and analytical characteristics, such as the depth resolution, characterized in that said optimized deposited  $Cs^0$  concentration is chosen only by adjusting the ratio ( $\tau = v_{er}/v_D$ ) between the erosion rate ( $v_{er}$ ) and the  $Cs^0$  deposition rate ( $v_D$ ), for a given sample and given primary bombardment conditions.

25 2. Method according to Claim 1, characterized in that said optimized deposited  $Cs^0$  concentration is continuously adjustable according to the relation :

$$c_{Cs} = \frac{1}{1 + (\tau - 1) \cdot \frac{\rho_M}{\rho_{Cs}} + \frac{\tau}{Y}},$$

30 wherein  $\rho_M$  is the density of the sample constituent,  $\rho_{Cs}$  the atomic density of the layer of Cs formed and Y the

**BEST AVAILABLE COPY**

11-01-2005

2

sputtering yield characterizing the primary bombardment conditions for the sample considered.

3. Method according to Claim 1 or 2, characterized in that the stream of Cs<sup>0</sup> is provided and 5 collimated in a column by means of :

- a temperature adjustment of an evaporator comprising a metallic cesium reservoir, and/or
- an aperture control of a motorized obturator located in the path of the cesium stream.

10 4. Method according to Claim 3, characterized in that the reservoir temperature range is maintained between 70 and 90 °C, corresponding to a pressure range from 1.10<sup>-4</sup> to 4.10<sup>-4</sup> mbar and in that the stability of the deposition rate is about 2% over 60 15 minutes.

5. Method according to anyone of Claims 1 to 4, characterized in that it is coupled to static or dynamic Secondary Ion Mass Spectroscopy (SIMS), preferably operating in the MCs<sub>x</sub><sup>+</sup> mode (x = 1, 2).

20 6. Method according to Claim 5, characterized in that the deposition rate of Cs<sup>0</sup> is continuously adjustable in the range from 0 to 10 Å/s, corresponding about to 0 - 4 monolayers per second.

7. Method according to anyone of Claims 1 to 25 4, characterized in that it is coupled to electron spectroscopy.

8. Method according to Claim 7, characterized in that electron spectroscopy is selected from the group consisting of Auger Electron Spectroscopy 30 (AES), Electron Energy Loss Spectroscopy (EELS), X-Ray Photoemission Spectroscopy (XPS) and Ultraviolet Photoemission Spectroscopy (UPS).

**BEST AVAILABLE COPY**

11-01-2005

3

9. Method according to Claim 1,  
characterized in that it further enables a stream of a  
chemical element other than Cs, evaporated under ultra-high  
vacuum, to create secondary emission for analytical  
purposes of  $M_1 M_2^{n+}$  clusters or  $M_2^{m-}$  ions or  $M_2^{m+}$  ions ( $n, m$   
5 integers) or electrons, wherein  $M_1$  and  $M_2$  are respectively  
the atoms or groups of atoms constituted by the chemical  
element other than Cs and the atoms or groups of atoms from  
the sample.

10 10. Method according to Claim 1,  
characterized in that the sole adjustable deposition rate  
of  $Cs^0$  or a chemical element other than Cs to an optimized  
value enables to optimize the intensity of secondary  
particles emitted by the sample.

15 11. Method according to Claim 1,  
characterized in that the useful yield, i.e. the  
sensitivity of the secondary emission species, preferably  
 $M^{m-}$ ,  $M^{m+}$ , and still more preferably  $Cs_x^{n+}$  and  $MCs_x^{n+}$ , is  
approximately optimized solely by adjusting said ratio ( $\tau$ ).

20 12. Method according to Claim 3,  
characterized in that the stream of  $Cs^0$  is automatically  
and continuously adapted via the obturator.

25 13. Energy and/or mass analyzer instrument  
for carrying out the method according to anyone of the  
preceding Claims 1 to 12, comprising a neutral cesium ( $Cs^0$ )  
deposition column, capable of delivering an adjustable and  
stable stream of pure neutral cesium, said neutral cesium  
column being usable simultaneously with a primary  
30 bombardment or a primary irradiation column, and comprising  
an evaporation block (1) including a reservoir (5) filled  
with pure metallic cesium, equipped with temperature  
control means (6,7), prolonged by a tube (2,11) up to a  
gun end piece (13) located close to the sample and equipped

**BEST AVAILABLE COPY**

11-01-2005

with beam collimation means (12), characterized in that said tube (2, 11) and gun end piece (13) equipped with beam collimation means (12) are further equipped with temperature control means (8, 14, 15) for preventing condensation and obturation risks.

14. Instrument according to Claim 13, characterized in that the evaporation block (1) is located in an external part which can be isolated from the main chamber (3) of the instrument by means of a gate valve (4) and capable of being separately pumped and vented.

15. Instrument according to Claim 13 or 14, characterized in that said beam collimation means comprise a motorized continuously adjustable obturator (12), preferably comprising a rotary disk using a slit of continuously variable width (16), said disk being driven by a stepper motor (17).

16. Instrument according to anyone of Claims 13 to 15, characterized in that, at the operation temperature, the neutral cesium ( $Cs^0$ ) is in liquid state and the evaporation block (1) lies with an inclination angle such as said liquid remains in the bottom of the reservoir (5) under gravity effect.

17. Instrument according to anyone of Claims 13 to 15, preferably a static or dynamic secondary ion mass spectrometry (SIMS) instrument, comprising a primary bombardment column and a secondary column equipped with secondary ion extraction means, a mass spectrometer, preferably of the type TOF (Time-Of-Flight), quadrupolar or with magnetic sector and ion detection means.

30

BEST AVAILABLE COPY